ESS Method 310.1:
Ortho-Phosphorus, Dissolved
Automated, Ascorbic Acid

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1.0 Scope and Application

1.1 This method may be used to determine concentrations of orthophosphate in most waters and wastewater in the range from 0.002-0.200 mg P/L. The concentration range may be extended to 0.2-2.00 mg P/L by utilizing a dilution loop.

1.2 Approximately 30 samples per hour can be analyzed.

2.0 Summary of Method

Ammonium molybdate and antimony potassium tartrate react in an acid medium with dilute solutions of orthophosphate-phosphorus to form an antimony-phospho-molybdate complex. This complex is reduced to an intensely blue-colored complex by ascorbic acid. The color is proportional to the phosphorus concentration.

3.0 Sample Handling and Preservation

Samples must be filtered through a 0.45 µm filter, cooled to 4°C and analyzed as soon as possible.

4.0 Interferences

4.1 Barium, lead, and silver interfere by forming a precipitate.

4.2 The interference from silica, which forms a pale-blue complex is small and can usually be considered negligible.

4.3 Arsenate is determined similarly to phosphorus and should be considered when present in concentrations higher than phosphorus.

5.0 Apparatus

Technicon AutoAnalyzer II system consisting of:

5.1 Sampler IV with a 30/h (2:1) Cam

5.2 Analytical manifold (orthophosphate in seawater) with internal heating bath at 37.5°C and dilution loop

5.3 Proportioning pump III

5.4 Colorimeter equipped with 50 mm flowcells and 880 nm interference filters

5.5 Printer/Plotter

6.0 Reagents

6.1 Stock Solution A; Sulfuric acid solution, 4.9 N: Add 136 mL concentrated H_2SO_4 to 800 mL Milli-Q water. Cool and dilute to 1 L with Milli-Q water.

6.2 Stock Solution B; Ammonium molybdate solution: Dissolve 40 g of $((NH_4)_6\ Mo_7O_{24}\bullet 4H_2O)$ in 900 mL Milli-Q water and dilute to 1 L. Store at 4°C.

6.3 Stock Solution C; Ascorbic acid: Dissolve 9 g of ascorbic acid ($C_6H_8O_6$) in 400 mL Milli-Q water and dilute to 500 mL. Store at 4°C. Keep well stoppered. Prepare fresh monthly or as needed.

6.4 Stock solution D; Antimony potassium tartrate: Dissolve 3.0 g of $(K(SbO)C_4H_4O_6\bullet \frac{1}{2}H_2O)$ in 800 mL Milli-Q water and dilute to 1 L. Store at 4°C.

6.5 Combined color reagent: Combine the following solutions in order, mixing after each addition: (Prepare fresh daily)

Stock A, 6.1 (4.9 N H_2SO_4)	50 mL
Stock B, 6.2 (Ammonium molybdate solution)	15 mL
Stock C, 6.3 (Ascorbic acid solution)	30 mL
Stock D, 6.4 (Antimony-tartrate solution)	5 mL

6.6 Water diluent solution: Add 4.0 g sodium lauryl sulfate and 5 g NaCl per L of Milli-Q water.

6.7 Stock phosphorus standard: Dissolve 0.4394 g of Potassium phosphate monobasic (KH_2PO_4) (dried at 105°C for one hour) in 900 mL Milli-Q water. Add 2 mL of concentrated H_2SO_4 and dilute to 1 L. 1.0 mL = 0.100 mg P (100 mg P/L).

6.8 Standard phosphorus solution 1: Dilute 100.0 mL of stock solution (6.7) to 500 mL with Milli-Q water. 1.0 mL = 0.020 mg P (20 mg P/L).

6.9 Standard phosphorus solution 2: Dilute 10.0 mL of stock solution (6.7) to 1 L. 1.0 mL = 0.001 mg P (1.0 mg P/L).

6.10 Working standard solutions:

6.10.1 Low Range (0.002-0.200 mg P/L): Prepare the following standards by diluting suitable volumes of standard solution 2 (6.9) to appropriate volumes with Milli-Q water:

mg P/L	mL of standard solution 2
0.005	1.0/200 mL
0.050	5.0/100 mL
0.100	50/500 mL
0.150	15/100 mL
0.200	40/200 mL

6.10.2 High Range (0.02-2.00 mg P/L): Prepare the following standards by diluting suitable volumes of standard solution 1 (6.8) to 200.0 mL with Milli-Q water:

mg P/L	mL of standard solution 1/200.0 mL
0.50	5.0
1.00	10.0
1.50	15.0
2.00	20.0

7.0 Procedure

7.1 Set up the manifold as shown in Figure 1. For the high concentration range, use the dilution manifold (Figure 1.).

7.2 Allow the colorimeter, and printer to warm up for 30 minutes. Obtain a stable baseline with all reagents, feeding Milli-Q water through the sample line.

7.3 Load the autosampler according to the CFDA Tray Protocol.

7.4 Analyze according to procedures in the LIMS-CFDA Methods Manual and General AutoAnalyzer Procedures.

8.0 Calculations

The phosphorus concentration is obtained directly from the LIMS plotter.

9.0 Precision and Accuracy

Precision and accuracy data are available in the Inorganic Chemistry Unit Quality Assurance Manual.

10.0 References

10.1 Methods for Chemical Analysis of Water and Wastes, U.S. Environmental Protection Agency, EPA 600/4-79-020, p 365.1, (1979).

10.2 Methods for Determination of Inorganic Substances in Water and Fluvial Sediments, U.S. Geological Survey Techniques of WaterResources Inv., Book #5, Ch.A1, p 514, (1985).

10.3 Ortho Phosphate in Water and Seawater, Industrial Method No. 155-71W, Technicon Instruments Corporation, Tarrytown, NY (1973).

Figure 1. Manifold Set Up